Precision

To estimate the precision of the method, five samples from the lot of heptachlor solution were independently assayed, giving a 95% confidence interval of 5.56 ± 0.065 mg. per ml. (±1.18%). This test was repeated in the same manner for the fertilizer, giving a 95% confidence interval of 2.81 ± 0.093 mg. per ml. (±3.31%).

At present, the method is being expanded to include simultaneous determination of several pesticides in the same solution, and results of this work will be published as soon as sufficient data have been accumulated.

Acknowledgment

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INSECTICIDE RESIDUES

Chromatographic Identification of Some Organophosphate Insecticides in the Presence of Plant Extracts

H. F. MacRAE[1] and W. P. McKINLEY

Food and Drug Directorate, Department of National Health and Welfare, Ottawa, Canada

Two paper chromatographic and detection methods are described for the identification of several organophosphate insecticides in the presence of plant extracts following cleanup on a cellulose-charcoal column. The methods are adequately sensitive for those organophosphates investigated and are relatively rapid and free from interference from plant extracts. These methods have been applied to the identification of only the parent compounds in the presence of apple, lettuce, cabbage, and orange extracts.

WIDESPREAD USE of organophosphate insecticides has created problems for the analyst who is concerned with the detection of undesirable amounts of insecticide residues in foodstuffs of unknown history. There is need for some relatively rapid scheme of qualitative analysis suitable for use as a guide to subsequent quantitative assessment. One approach to this problem would be through a study of the chromatography of plant extracts with the ultimate aim of being able to predict the separation of the insecticide residues from the known chromatographic behavior of the pure substances (*8*). A major problem here is the separation of the insecticide from naturally occurring plant substances that may interfere either with the chromatographic separation or with the final identification tests.

Chillwell and Hartley (*2*) have reviewed the methods for the determination of residual organophosphorus insecticides in foodstuffs. Several paper chromatographic methods for the identification of the pure compounds have been reported (*3–6, 10–12*), but these methods have not been applied generally to the practical problem of identification of organophosphate insecticides in the presence of plant extracts. Recently, Laws and Webley (*9*) reported a method for the determination of organophosphorus insecticides in vegetables, which consists in extraction of the insecticides when added to plant material, separation into petroleum-soluble and water-soluble groups, chromatography of the groups on alumina and activated carbon, respectively, and subsequent determination of the phosphorus by measurement of the molybdenum-blue complex.

The object of a cleanup procedure is to reduce the contribution made to the final determination by natural plant products. The degree of refinement necessary in cleaning up extracts from possible interfering substances, before the final determination, depends to a large extent on the selectivity and sensitivity of the final procedure, the nature of the crop, and the lowest level of insecticide that must be detected with certainty (*2*).

Many cleanup procedures described in the literature are applicable to one type of crop or one specific pesticide. In the present study, a cleanup procedure was sought which would be applicable to paper chromatographic identification of a number of organophosphate insecticides in the presence of a wide variety of plant materials. The methods described have been applied for identification of only the parent compounds when added to apple, lettuce, cabbage, and orange extracts obtained by two extraction procedures. These crops represent waxy plants, leafy vegetables, and citrus fruits, respectively.

Experimental

Apparatus. Chromatographic columns, 40 × 2.5 cm., fitted with stopcock, coarse, fritted-glass disk, and solvent reservoir (ca. 200 ml.) at the top.

Chromatographic tanks. Museum jars, size 11 without specimen supports, Arthur H. Thomas Co., Philadelphia, Pa. Cylindrical jars with covers, size 6 × 18 inches, Canadian Laboratory Supplies Ltd., Montreal.

Chromatographic spray bottles, 50-ml. capacity.

Standard micropipets, graduated in μl. and equipped with a safety pipet filler (Pripipette Instrumentation Associates, New York, N. Y.).

Reagents. All chemicals were analytical reagent grade, and all solvents were redistilled before use.

SOLKA-FLOC. Highly purified wood Cellulose, BW 40 (Brown Co., Boston, Mass.) extracted twice with freshly distilled acetone.

ACTIVATED CHARCOAL. Darco G 60 (Brickman Co., Montreal, Canada).

STANDARD SOLUTIONS. Acetone solutions of the organophosphate insecticides were made up to contain 2 mg. per ml. of solution, and appropriate dilutions were used.

CHROMATOGRAPHIC SYSTEM A. Whatman No. 1 filter paper sheets $8\frac{1}{2} \times 8\frac{1}{2}$ inches; immobile phase, 10% (v./v.) 2-phenoxyethanol in ethyl ether; mobile phase, isoöctane (2,2,4-trimethyl pentane) (*11*).

CHROMATOGRAPHIC SYSTEM B. Acetylated paper sheets 4 × $16\frac{1}{2}$ inches 40 to 45% acetyl (CH_3CO-), Carl Schleicher-Schull, Dassel, Kreis Einbeck, West Germany; immobile phase, 2% (v./v.) U.S.P. mineral oil (light) in ethyl ether, mobile phase, 70% aqueous acetone (*10*).

[1] Present address: Department of Animal Science, Macdonald College, McGill University, Quebec, Canada.

Table I. Compounds Studied

Common or Trade Name	Chemical Name	Chromatographic System[a]	Detection Reagent[b]
Co-Ral	*O*-3-chloro-4-methylumbelliferone *O,O*-diethyl phosphorothioate	B	FCSSA
Diazinon	*O,O*-diethyl *O*-(2-isopropyl-4-methyl-6-pyrimidyl) phosphorothioate	B	FCSSA
Di-Syston[c]	*O,O*-diethyl *S*-2-(ethylthio)ethyl phosphorodithioate	A B	IOP FCSSA or IOP
EPN	*O*-ethyl *O*-*p*-nitrophenyl phenylphosphonothioate	B	FCSSA
Ethion	*O,O,O',O'*-tetraethyl *S,S'*-methylene bisphosphorodithioate	A	IOP
Guthion	*O,O*-dimethyl *S*-(4-oxo 1,2,3-benzotriazin-3-ylmethyl)phosphorodithioate	B[d]	FCSSA
Malathion	*S*-(1,2-bis(ethoxycarbonyl)ethyl)-*O,O*-dimethyl phosphorodithioate	A	IOP
Nemacide	*O*-2,4-dichlorophenyl *O,O*-diethyl phosphorothioate	B	FCSSA
Parathion	*O,O*-diethyl *O*-*p*-nitrophenyl phosphorothioate	B	FCSSA
Rogor	*O,O*-dimetnyl *S*-(*N*-methylcarbamoylmethyl) phosphorodithioate	B[d]	FCSSA or IOP
Systox[c]	*O,O*-diethyl *O*-2-(ethylth o)ethyl phosphorothioate	A	IOP
	O,O-diethyl *S*-2 (ethylthio)ethyl phosphorothioate	B	FCSSA or IOP
Thimet[c]	*O,O*-diethyl *S*-(ethylthio)methyl phosphorodithioate	A B	IOP FCSSA
Trithion[c]	*S*-(*p*-chlorophenylthio)methyl *O,O*-diethyl phosphorodithioate	A B	IOP FCSSA

[a] System A, 2-phenoxyethanol-isoöctane, using Whatman No. 1 filter paper. System B, Mineral oil–70% aqueous acetone, using acetylated paper.
[b] FCSSA, Ferric chloride–salicyl sulfonic acid reagent. IOP, Iodoplatinate reagent.
[c] These compounds may be detected equally well in either System A or B using the detection reagents indicated.
[d] These compounds may also be chromatographed in the methanol–water–ammonia system (*10*) and detected with IOP.

DETECTION REAGENT. Iodoplatinate reagent (IOP) (*7*). To prepare the stock solution, dissolve 1 gram of platinic chloride in 10 ml. of H_2O and add this solution to a solution containing 10 grams KI dissolved in 250 ml. of H_2O. This solution is stable indefinitely.

The working solution is prepared by mixing 1 volume of the stock solution with 6 volumes of distilled water when this reagent is used on Whatman No. 1 paper (Chromatographic System A). This diluted reagent is also stable indefinitely. When acetylated paper is used (Chromatographic System B), the working solution is prepared by mixing 1 volume of the stock solution with 3 volumes of ethyl alcohol. This solution is unstable and must be prepared immediately before use.

FERRIC CHLORIDE–SALICYLSULFONIC ACID REAGENTS (*10*).

BROMINE VAPORS. A concentrated aqueous solution of Br_2 placed in the bottom of a large glass tank.

FERRIC CHLORIDE SOLUTION. Dissolve 0.1 gram $FeCl_3.6H_2O$ in 1 ml. of 1*N* HCl and dilute to 100 ml. with 80% (v./v.) ethyl alcohol.

SALICYLSULFONIC ACID SOLUTION. Dissolve 1 gram of the British Drug House reagent in 80% (v./v.) ethyl alcohol.

Staining with these reagents will be referred to as staining with FCSSA.

Preparation of Plant Extracts. Two methods of extraction were employed to test the effectiveness of the cleanup procedure. The first was based on that described by Anglin and McKinley (*1*) for DDT and related pesticides. This is a vigorous extraction, and any procedure which would remove the large amount of plant extractives other than pesticides would be likely to clean extracts prepared in other ways. The second method was considered as suitable for the extraction of organophosphate insecticides from plant materials. The extraction procedure was as follows:

Blend 500 grams of plant material for 1 minute in a Waring Blendor with 300 ml. of acetone. Add another 500 grams of plant material and blend for 1 minute. Add 200 ml. of acetone and blend for 2 minutes more. Filter the material through cheesecloth and centrifuge the filtrate. Blend the plant residue for 2 minutes with 1000 ml. of benzene. Filter the material through cheesecloth and centrifuge the filtrate. Keep the aqueous acetone and benzene solutions separate. Extract the aqueous acetone phase with 300 ml. of chloroform followed by two extractions, each with 100 ml. of chloroform. Combine the chloroform and benzene extracts.

Cleanup Procedure. An appropriate portion (100 to 200 ml. representing 66.6 to 133.3 grams of plant material) of the extract was placed in a beaker, and a known amount of the standard insecticide solution added (a range representing 0.1 to 0.5 p.p.m. of plant tissue). The extract was evaporated to dryness in a water bath maintained at 35°C. under stream of filtered air.

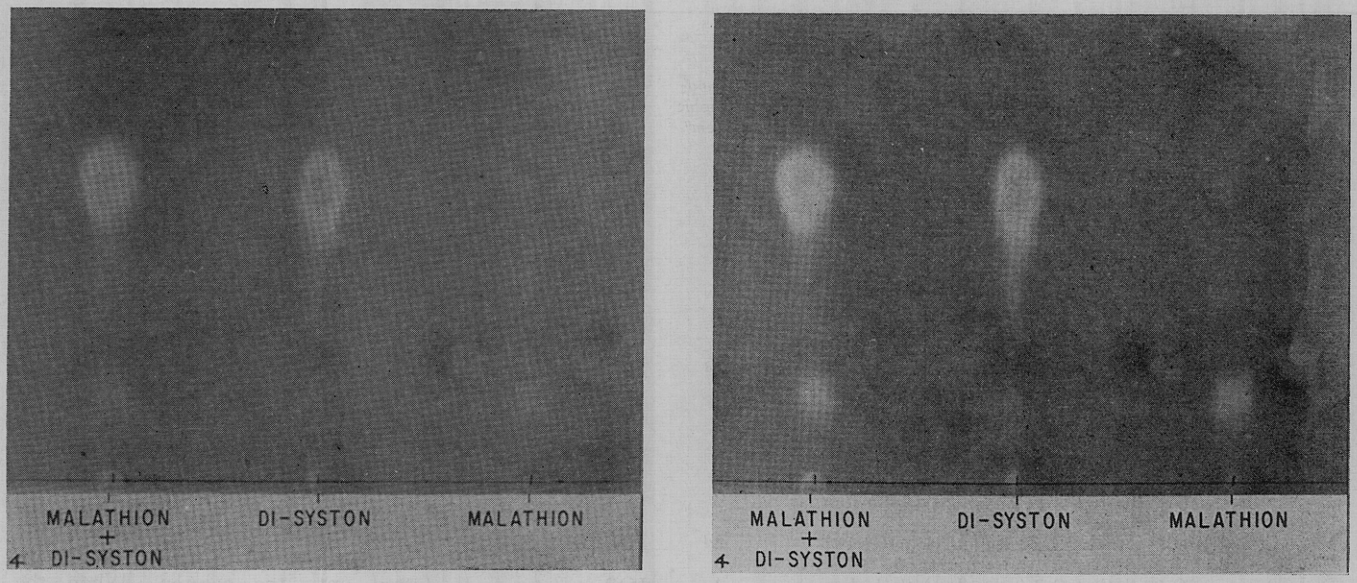
The column material was prepared by mixing in the dry state 25 grams of Solka-Floc and 5 grams of Darco G 60. Approximately 200 ml. of acetone was added to the mixture to form a relatively thin slurry which was then poured into the column. Excess acetone was drained from the column, but the top of the column was not allowed to dry.

The residue from the evaporated plant extract was dissolved in 5 ml. of acetone. It was important to wash the walls of the beaker with acetone and to scrape the walls and bottom of the beaker with a glass rod to ensure adequate dissolution of the residue. When the excess acetone had just passed through the top of the column, the dissolved residue was transferred onto the column. When the solution of residue had just passed into the column, a 25-ml. graduated cylinder was placed under the column to collect the eluate. This transfer procedure was repeated twice using 5-ml. portions of acetone. The sides of the column were washed with a small portion of acetone. When the latter had passed into the top of the column, 150 ml. of acetone was added to the reservoir. The first 25 ml. of eluate was discarded, and the subsequent 125 ml. was collected. This fraction contained all the organophosphates studied. The eluate was transferred, in portions, to a 50-ml. beaker and evaporated almost to dryness as described for the plant extracts. The residue in the beaker was then transferred to a small test tube (1 × 8 cm.) with four small portions of acetone, washing the walls of the beaker with each portion, and then evaporated to a volume of approximately 50 μl. The residue was then transferred to the chromatogram with a microliter pipet. This transfer procedure was repeated three times using 25 μl. of acetone for each transfer.

Chromatography. Chromatographic analyses were carried out in either System A or System B or in both. Paper sheets were impregnated with the immobile phase as described by MacRae and McKinley (*10*). Table I indicates the compounds studied and the chromatographic systems and detection reagents which were the most satisfactory for the identification of each compound. It is, of course, advisable to use two chromatographic systems if identification is uncertain in any one system.

Results and Discussion

Chromatography of Pure Compounds. Figure 1 shows a typical chromatogram developed in System A and stained with IOP. Malathion is unique among all the thiophosphates studied in that considerable time is necessary for a complete reaction with



(A) Photographed 1 hour after staining with IOP reagent

(B) Same chromatogram photographed 12 hours after staining

Figure 1. Typical chromatogram developed in System A

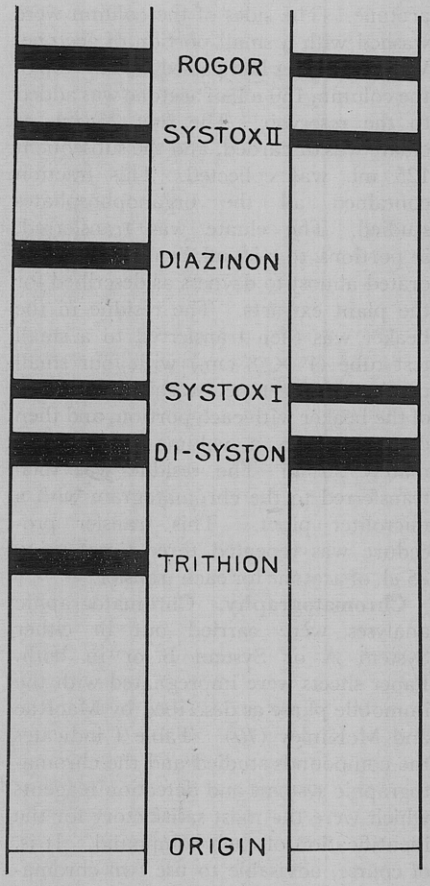


Figure 2. Diagram of a typical chromatogram developed in System B

(The left-hand portion of this chromatogram was stained with FCSSA reagent. The right-hand portion was stained with IOP reagent.)

IOP. This time factor affords a useful means of detecting malathion in the presence of other thiophosphates. The IOP reagent is very sensitive for the detection of Di-Syston, ethion, Guthion, malathion, Rogor, Systox, Thimet, and Trithion on Whatman No. 1 paper, i.e., those thiophosphates which contain more than one sulfur atom in the molecule. It is not satisfactory for the detection of one-sulfur compounds, e.g., Co-Ral, Diazinon, EPN, Nemacide, and parathion, either on Whatman No. 1 paper or acetylated paper. It is possible to detect only Di-Syston, Systox, and Rogor on acetylated paper using the IOP reagent.

Figure 2 shows a diagram of a typical chromatogram developed in System B. Samples were applied in bands to permit a better separation of compounds which have closely related mobilities in this solvent system. Chromatograms were run three times for 11 hours each time, with thorough drying of the chromatogram between each run. This redevelopment technique has been applied by Pasieka (*13*) to the separation of amino acids on paper chromatograms and by Prusikova (*14*) to similar separations of steroids. The chromatogram was cut longitudinally into 2-inch strips, and the part shown on the right of Figure 2 was sprayed with the IOP reagent and the part on the left with the FCSSA reagent. Twenty-five micrograms of each compound was applied to the whole chromatogram. Chromatography of organophosphates on acetylated paper (System B) has provided the most effective method, of the many chromatographic systems studied, for separating a large number of compounds on any one chromatogram. This is particularly true when the solvent redevelopment technique is used. Furthermore, FCSSA reagent on acetylated paper makes it possible to detect all the thiophosphates studied and to distinguish thiophosphates from other organophosphates on the same chromatogram (*9*).

Application of the Method to Crop Extracts. The isopropyl alcohol–benzene extraction (*7*) of plant material yielded large amounts of extraneous plant material. For this reason, this extraction procedure was employed to check the effectiveness of the Solka-Floc–Darco G 60 column for the separation of pesticides from other plant extractives. Various proportions of the two column materials were tested, and 25 grams of Solka-Floc mixed with 5 grams of Darco G 60 provided adequate cleanup of the extracts and permitted the elution of the organophosphates for chromatographic identification. Smaller amounts of the column materials resulted in the elution of a white, waxy material and some pigment along with the organophosphates, particularly from apple extracts. If the column was prepared as described above, and the first 25 ml. of eluate was discarded, all the organophosphates which could be recovered by this method appeared in the succeeding 125 ml. of eluate.

Figure 3 shows a typical chromatogram of an isopropyl alcohol–benzene extract of apples, to which had been added 0.2 p.p.m. of Di-Syston and 0.15 p.p.m. of malathion. The absence of any detectable spots in the crop blank demonstrates that there was no interference from extraneous plant material after cleanup on the column.

The aqueous acetone-benzene extraction procedure was employed because it is a more suitable method for extracting organophosphates from plant material. Most organophosphate insecticides are in nonionic form and are favorably partitioned into chloroform from aqueous solutions because of the exceptionally strong molecular forces between P→O and $CHCl_3$, or because the cor-

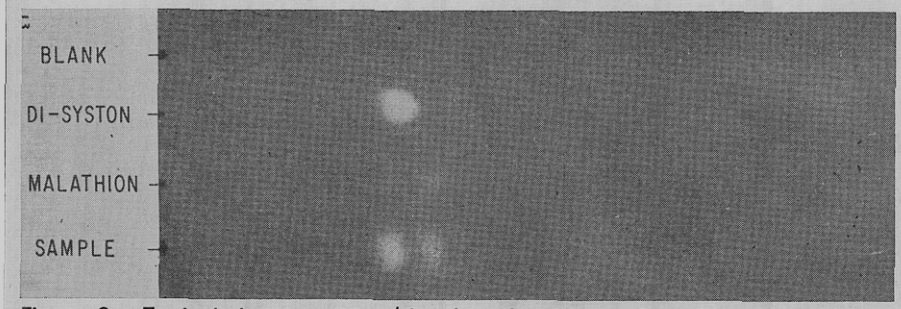


Figure 3. Typical chromatogram (developed in System B and stained with IOP reagent) showing:
an isopropyl alcohol–benzene extract of apples to which had been added 0.2 p.p.m. of Di-Syston and 0.15 p p.m. malathion (Sample), reference malathion, Di-Syston, and a crop blank.

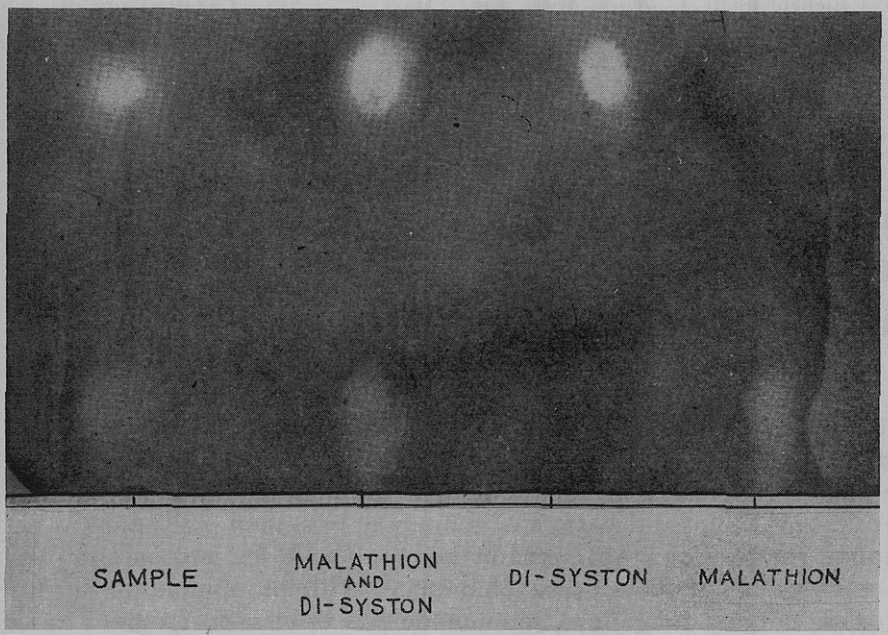


Figure 4. Typical chromatogram (developed in System A and stained with IOP reagent) showing:
a chloroform-benzene extract of lettuce to which had been added 0.2 p.p.m. Di-Syston and 0.15 p.p.m. malathion (Sample), reference malathion, and Di-Syston chromatographed singly and combined

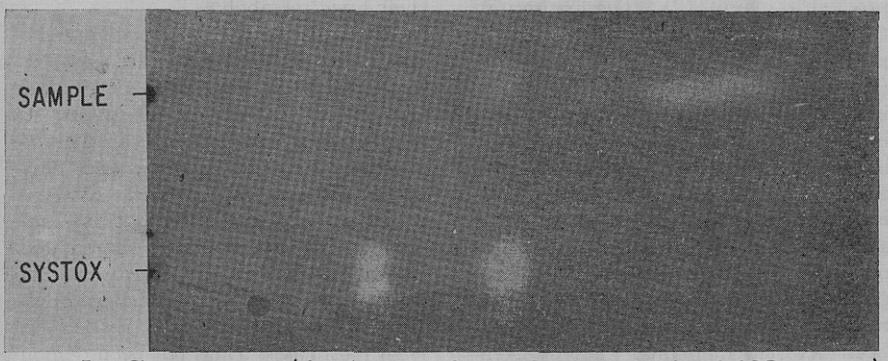


Figure 5. Chromatogram (developed in System B and stained with IOP reagent) showing:
Systox which had been added to an aged chloroform-benzene extract of cabbage (Sample) and reference Systox

Table II. Recovery of Compounds Added to Crop Extracts[a]

Crop	Compounds Recovered	Compounds Not Recovered	Compounds Converted
Apple	Diazinon	EPN	Ethion
	Di-Syston		Guthion
Lettuce	Malathion	Nemacide	
	Rogor		Trithion
Cabbage	Systox	Parathion	Co-Ral
Orange	Thimet		

[a] All compounds listed were added to two types of extracts for each crop at 0.1, 0.5, and 1.0 p.p.m.

responding P → S compounds are much less hydrophilic (*2*). Derivatives produced in the plant, which are generally oxidation products, are more hydrophilic, and therefore more exhaustive extraction with chloroform is needed when determination of these derivatives is in question.

Partitioning from aqueous acetone into chloroform did not suffice, in the present study, to reduce the level of interfering substances to permit chromatographic identification of the compounds added to the extracts. It was therefore necessary to pass the aliquots from the combined chloroform-benzene extract through the column to effect further cleanup prior to chromatography.

Figure 4 shows a typical chromatogram of a chloroform-benzene extract of lettuce to which malathion and Di-Syston, at 0.15 and 0.2 p.p.m., respectively, were added.

Table II summarizes the compounds which were added to various crop extracts and those which were recovered, not recovered, or converted during the procedure, according to the chromatographic and detection methods employed. Conversion products, where they occurred, were not identified.

Three sources of contamination encountered during the course of this work interfered seriously with the identification of compounds by the two detection reagents. Firstly, traces of oil in the air-line (used for evaporation of solvents) gave spots on the chromatogram in the regions where the insecticides were located. It was necessary to install a filter on the air-line to avoid this contamination. Secondly, reagent grade acetone used for column elution contained contaminants which interfered seriously in the chromatography, especially when System B and the FCSSA reagent were used. Therefore, the acetone must be redistilled before use. Thirdly, the purified wood cellulose used in the column contained contaminants which were soluble in acetone and which interfered with the chromatography, particularly in acetylated paper (System B). It was necessary, therefore, to pre-extract the Solka-Floc with acetone.

One further difficulty arose in the recovery of pesticides added just prior to cleanup from extracts stored at room temperature for 2 to 3 weeks. Several of the compounds which could be recovered from fresh extracts, were either converted or destroyed when added to extracts which had been prepared some time previously. Figure 5 shows a typical result obtained with Systox which had been added to a cabbage extract prepared 3 weeks before further examination. Systox could be recovered unchanged when added to freshly prepared extracts.

Finally, although this work has not been extended as yet to actual analyses

of insecticide residues, the method shows sufficient promise to warrant such an extension. Furthermore, these procedures may possibly be applied to the identification of degradation products of the organophosphates formed in the plant.

Acknowledgment

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INSECTICIDE RESIDUES

The Detection of Residues of Systox and Its Toxic Metabolites in the Presence of Other Organophosphorus Pesticides

J. M. ADAMS, C. A. ANDERSON, and DANIEL MacDOUGALL

Chemagro Corp., Kansas City, Mo.

A method has been developed for detection of residues of Systox and its metabolites in plants. The method is based upon chromatographic separation on paper and subsequent characterization through the use of the color forming agent, 2,6-dibromo-N-chloro-p-quinoneimine. The method has a sensitivity for detection of 0.3 p.p.m. based on a sample of 100 grams and will distinguish residues of Systox and its metabolites in the presence of other organophosphorus pesticides and cholinesterase inhibitors. With the application of a preliminary chromatographic cleanup procedure, the method has been used for the detection of Systox residues in a large number of crops.

THE DETERMINATION of Systox (demeton) residues in plant material has been based in the past on the measurement of cholinesterase inhibition (*8*). In view of the numerous compounds presently registered as pesticides which are either cholinesterase inhibitors themselves or are capable of being converted into cholinesterase inhibitors, this method possesses insufficient specificity to be useful for determining the presence or absence of Systox in plant material of unknown history. The work described in this paper was carried out to develop a detection method for Systox residues in the presence of residues of other cholinesterase-inhibiting pesticides or their metabolites.

The commercial product, Systox (Chemagro Corp.), is a mixture of two insecticidally active isomers: *O,O*-diethyl *O*-2-(ethylthio) ethyl phosphorothioate (I); and *O,O*-diethyl *S*-2-(ethylthio) ethyl phosphorothioate (II). The isomers are present in the commercial material in a ratio of approximately 2 to 1.

Extensive metabolic studies on Systox have been conducted by Metcalf and coworkers (*5*, *7*, *9*, *12*). These studies have shown conclusively that the Systox isomers do not persist as such for more than 1 or 2 weeks after application of the product to plants (*12*). The first step in the metabolism of the thiono isomer in plants is its conversion to the sulfoxide (*5*, *9*). Also, when the thiono isomer is applied to cotton plants, there is an appreciable residue of the thiono isomer sulfone present, even 10 days after treatment (*7*). The thiol isomer is likewise oxidized at the side chain sulfur atom to form both the sulfoxide and the sulfone (*6*). The phosphate derivatives which might be formed from the thiono compounds have not been detected in plants. This is likely due to their rapid rate of hydrolysis. Thus, it is apparent that a generally applicable method for Systox residues should determine not only the parent isomers but their sulfoxides and sulfones as well. The method described here will identify the Systox thiol isomer and the sulfoxide and sulfone of both isomers. The thiono isomer cannot be distinguished from other interfering pesticides by this method.

Compounds listed in Table I are registered as cholinesterase inhibitors. A specific method for Systox must be capable of detecting Systox and its metabolites in the presence of these cholinesterase-inhibiting compounds and/or their toxic metabolites.

The organophosphorus compounds containing the P=S group are converted in the animal body to their oxygen analogs (*3*) containing a P=O group. In this way, phosphorodithioates are metabolized to phosphorothioates, and phosphorothioates to phosphates. Evidence continues to accumulate that the anticholinesterase activity of compounds containing the P=S group is due to their conversion to the oxygen analogs in the mammalian liver (*3*).

In most cases, it has not been demonstrated that the oxygen analog of the compounds listed in Table I are formed in plants. However, Metcalf *et al.* (*13*, *15*) have shown that Di-Syston (trademark of Farbenfabriken Bayer) derivatives containing the P=O group are formed in both cotton and tomato plants. In view of these observations, there is a definite possibility that traces of the oxygen analogs of the compounds in question may be present in plants. Therefore, all of the oxygen analogs of these compounds were considered as possible interferences in the detection of Systox residues.

It has been reported (*14*) that Tri-